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TECHNICAL REPORT 2093

DEVELOPMENT OF A NEW IGNITION TEMPERATURE APPARATUS

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NOVEMBER 1954

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DOVER, N. J.

ORDNANCE PROJECT TA3-5101
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CORRECTION

Picatinny Arsenal Technical Report 2093

Development of a New Ignition Temperature Apparatus

by

James E. Abel
Pfc. Jack Alster
Arthur C. Forsyth
Henry Jackson
Charles Pataky

November 1954

The Table on page 4, Dextrinated Lead Azide, .002 gm should be corrected to read as follows:

Dextrinated Lead Azide, .002 gm

10	329.4	1.528	0.218
15	335.0	1.050	0.040
10	346.0	0.807	0.060
12	360.0	0.537	0.052

for

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(DEVELOPMENT OF OPTIMUM EXPLOSIVE TRAINS)

DEVELOPMENT OF A NEW IGNITION TEMPERATURE APPARATUS

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Approved:

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OBJECT

To develop an instrument to determine the ignition temperature and time to ignition of one milligram or less of primary explosive.

ABSTRACT

This is the second progress report on the "Development of Optimum Explosive Trains" and is concerned with the development and utilization of an electronic device to determine explosion temperatures and ignition times of one milligram samples of primary explosives. With this instrument the time to ignition can be determined to ± .001 seconds and the explosion temperature to ± 2°F. The activation energies calculated from data obtained with this instrument in the .5 to 10 second interval are shown below:

Compound	Activation Energy, E. k cal/mole	$t = B_e^{\frac{B}{RT}}$
Mercury Fulminate	29.81	- 13.85
Lead Azide	23.74	- 8.43
Basic Lead Styphnate	75.39	- 28.20
KDNBF	82.58	- 34.12
Hexammine Chromic Perchlorate	29.82	- 9.88

INTRODUCTION:

1. The work on Project TA3-5101 entitled "Development of Optimum Explosive Trains" has been concerned with the synthesis of new primary explosives (Ref B). These compounds previously have been assessed for their explosive properties by tests standard at the Arsenal (Ref C). One of these, the explosion temperature test (Ref C) is the subject of this report and consists of varying the temperature of a molten metal bath between determinations in such a way as to have ten flashes or explosions over an induction time interval of 2 to 10 seconds. A time vs temperature curve is plotted and the five-second explosion temperature is interpolated from the graph. The data is obtained by immersing a No. 8 blasting cap containing 0.01 of primary explosive or 0.02 gm high explosive into a molten metal bath of known temperature. The time to ignition is determined with a stop watch. A total of from 0.5 gm to 5.0 gms of sample is necessary to complete one explosion temperature test. Since the accuracy of the stop watch method can vary from .2 to 0.4 seconds it was believed advisable to adopt a more precise timing method. It also seemed advisable to replace the molten metal bath by an electrical heating unit which would bring the explosive to the desired temperature in a shorter time. In addition, it seemed advantageous to construct an instrument that would determine a complete explosion temperature curve and would require only 0.1 gm sample of explosive.

2. Studies have shown (Refs D, E, and G) that the activation energies of explosive compounds may be calculated from the above mentioned explosion temperature curves. Since the activation energy of a compound is known to be related to the case of electrical initiation of the compound (Ref H) and to its stability (Ref D), it seemed advisable to calculate this value for standard primary compounds as well as new compounds synthesized in this program (Ref B).

DISCUSSION OF RESULTS:

3. The instrument designed for precise ignition temperature and time for ignition measurements consists of a small electrically heated plate with a centrally located iron-constantin thermocouple for determining its temperature, (Plate 1) and a photoelectric optical system for determining time to ignition. Current accurate to 0.10 amperes is used to heat the plate and the temperature of the plate is determined by the E M F output of the thermocouple with a Leeds and Northrup Potentiometer (Model 3662). Thus, when the explosive is dropped on the plate the temperature for ignition is definitely known. To determine the time for ignition at a fixed temperature a beam of light (1/16" to 1/8" diameter) is focussed across the plate to a Photomultiplier (Model 1P 21) (Plates 1 and 2). When one milligram

of the compound is dropped on the plate the light beam is interrupted causing the phototube to produce a positive electrical pulse which is amplified and in turn starts the Electronic Counter Chronograph. When the substance detonates, the light from the flash again causes the phototube to conduct, generating a negative pulse. This negative pulse is fed through the amplifier and inverted to a positive pulse to trigger the stop circuit of the Potter Chronograph. Times over one second are recorded by the Veeder Root Counter connected to the last decade of the Chronograph.

4. Explosion temperature curves were obtained on three primary explosives and two new experimental compounds with this new instrument. (Fig 1). The five-second ignition temperatures for these compounds and data determined with the molten bath method (Ref C) are shown below:

Compound	Molten Metal Bath Method, 0.010 gm	New Method, grams
Mercury Fulminate	210°C	209 (.001gm)
Basic Lead Styphnate	295	293 (.0025)
Lead Azide (dextrinated)	340	* (.002)
Potassium Dinitrobenzofuroxan	250	245 (.001)
Hexamaine Chromic Perchlorate	350	346 (.002)

* 100% ignition did not occur with 2 mg sample at 5 seconds

5. The main feature of this instrument is the precision of the data which can be obtained. At least ten ignition determinations were made at each temperature and the standard deviation for each point determined. The data for Mercury Fulminate and Dextrinated Lead Azide are shown below:

Mercury Fulminate, .001 gm

Number Tested	Temperature °C	Induction Time, Seconds	Standard Deviation/ Seconds
10	203	7.115	0.290
12	210	4.506	0.017
10	215	3.333	0.146
10	220	2.408	0.206
10	226	1.702	0.287
10	233	1.098	0.080
	Dextrinated Lead Azide, .002 gm		
10	329.4	.537	0.059
15	335.0	.807	0.060
10	346.0	1.05	0.040
12	360.0	1.528	0.218

The above standard deviations are typical of the precision of the data in this report. Also, the standard deviation is equal to or better than the most recent values for this type of determination (Ref E).

6. Arrhenius was among the first to show the large effect of temperature on the rates of chemical reactions (Ref D). Several workers (Ref D, G, E, and I) studied the effect of temperature on the times to ignition or induction periods of the pre-ignition reactions of several explosives. Semenoff (Ref I) expressed the relationship between the induction period and ignition temperature as follows:

$$t = B_e \frac{E}{RT}$$

where t = induction period in seconds
E = activation energy K cal/mole
R = gas constant
B = a constant
T = temperature, °K

putting the equation in the form:

$$\log t = \log B + \left[\frac{E}{R} \log_{10} e\right] \frac{1}{T}$$

it is seen that a straight line is obtained when $\log_{10} t$ is plotted against $\frac{1}{T}$ with slope equal to $\frac{E}{R} \log_{10} e$. The activation energy is therefore given by the product of the slope and $\frac{R}{\log_{10} e}$. The slopes for the various explosives studied in this report were determined by the method of least squares and the activation energies calculated therefrom.

7. The activation energies of Mercury Fulminate, Dextrinated Lead Azide and Basic Lead Styphnate were calculated from data determined to plot their explosion temperature curves (Figs 1 and 2). The values obtained by the new method and reported values of other workers are shown below:

Determined Values

Compounds	Weight Sample gm	Activation Energy K cal/mole	B
Mercury Fulminate	.001	29.81	- 13.85
Basic Lead Styphnate	.002	75.39	- 28.20
Lead Azide	.002	23.74	- 8.43

Values of Previous Workers

Mercury Fulminate (Ref D)	.001	33.4	- 14.38
Lead Azide (Ref D)	.003	23.4	- 8.2

The Activation Energies of Hexammine Chromic Perchlorate and Potassium Dinitrobenzofuroxan were also obtained (Figs 1 and 2). The results are shown below:

Compounds	Weight Sample gm	Activation Energy K cal/mole	B
Potassium Dinitrobenzofuroxan	.001	82.58	- 34.62
Hexammine Chromic Perchlorate	.002	29.82	- 9.88

8. It is to be noted that for all compounds straight line graphs are obtained when the logarithms of the induction periods are plotted against the reciprocals of the absolute ignition temperatures in the range of .5 to 10.0 seconds. This is in accordance with the Semenoff Equation (Ref I).

Inasmuch as the above graphs all involve induction periods greater than 0.5 second it seemed of interest to determine whether an extrapolation of the straight lines to shorter time intervals such as has been done by A. R. Ubbelohde (Ref D) is permissible. If so, the validity of the Semenoff Equation would be further strengthened and predictions about high ignition temperatures and short induction periods would be justified. A study of KDNBF in the range of 7.57 to 0.0107 seconds corresponding to 246°C and 351°C, respectively, revealed however a parabolic shape of the plot (Fig 3). The relation which has been considered before as a linear proportionality above the 0.5 second point now appears to be an approximately linear segment of a parabola. Although this fact does not seriously affect the value of the activation energy (previously obtained for potassium dinitrobenzofuroxan from the slope of the linear plot), it must nevertheless be said that the activation energy so calculated, refers to simple pre-ignition reactions occurring at temperatures characteristic of times greater than 0.5 second. At higher temperatures or shorter induction periods, it seems that we are no longer dealing with a simple pre-ignition reaction whose activation energy is uniquely determined by the Semenoff Equation. Rather, we may have a complex series of reactions and interactions between decomposition product molecules, as well, whose overall activation energy can not be obtained by such a simple relation. In such cases an extrapolation of a linear plot to the short induction period range is not valid and one must have a knowledge of the shape of the entire curve before predictions about high ignition temperatures and low induction periods can be made, at least in the case of KDNBF.

9. It is intended to carry out an intensive further investigation into the low induction period region for KDNBF and the other compounds, as well, to determine whether the parabolic shape of the curve is a general phenomenon. Apparatus similar to that employed in the work above but modified so as to attain induction intervals in the one-tenth millisecond region, has, in fact, been constructed. It is anticipated that the new results will soon be forthcoming.

EXPERIMENTAL PROCEDURE

10. The operation of the explosion ignition time consists of electronic systems which control the temperature of the ignition plate and the timing mechanism.

a. The temperature of the ignition plate is set by varying the current through a No. 18 evanohm wire. A fine and coarse adjustment of the latter regulates the current to ± 0.0 ampere over a range of 0 - 10 amperes. After the system has come to equilibrium,

the temperature of the plate is determined by reading the EMF output of the B & S No 30 iron-constantan thermocouple on the Leeds and Northrup Potentiometer (Model 8662).

b. The timing mechanism involves utilization of the photoelectric effect. As shown in Plate 1 a beam of light is passed over the heating plate and on to the phototube. Upon dropping of the explosive sample on the heating plate, the beam is interrupted and an electronic counter chronograph and second counter triggered. The latter counts until the instant of explosion when a flash again actuates the phototube and now stops the counters. A direct reading of the induction period is thus obtained. It is essential that the beam of light be of small diameter and centered over and as close to the plate as possible. A variable lens opening in (C) controls the beam width and a platform on which the light source (B) is mounted provides for horizontal and vertical displacements of the light beam. The phototube sensitivity to changes in light intensity should be at an optimum. Plate 2, B, illustrates the appropriate instrument control.

11. Once it is established that the timing system is working efficiently and the temperature of the plate is constant, the time for ignition at that temperature may be determined. One milligram of sample is weighed on a Roller-Smith Milligram balance and dropped through the funnel on to the plate. The seconds are read on the Veedar Root Counter and the milliseconds on the Electronic Counter Chronograph (Model 450A). The funnel is then removed and the plate cleaned with an electrically driven steel wool brush. The temperature is allowed to come to equilibrium again and the determination repeated. Usually a five minutes waiting period is required for the plate to come to equilibrium with the surroundings. If the material blackens the glass slides (Plate 1) clean ones must be inserted so that the path of light beam will not be obstructed.

12. After 10 to 20 ignition times have been determined at one temperature, the temperature is raised by increasing the current and ten or more ignition times are determined. The average of the times is taken as the time. Usually four to six points are necessary to plot a curve.

ACKNOWLEDGMENT:

The advice of Lieutenant J. E. Boyd of the Naval Research Laboratory in the early stages of development of this instrument, is

hereby acknowledged. Also, the co-operation of the Instrumentation Unit and particularly Mr. E. Hazelhurst and Mr. L. Ouelette in the design of this instrument is also hereby acknowledged.

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INCLOSURES:

1. Figures 1, 2, and 3
2. Plates 1, and 2

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FIGURE I

EXPLOSION TEMPERATURE CURVES

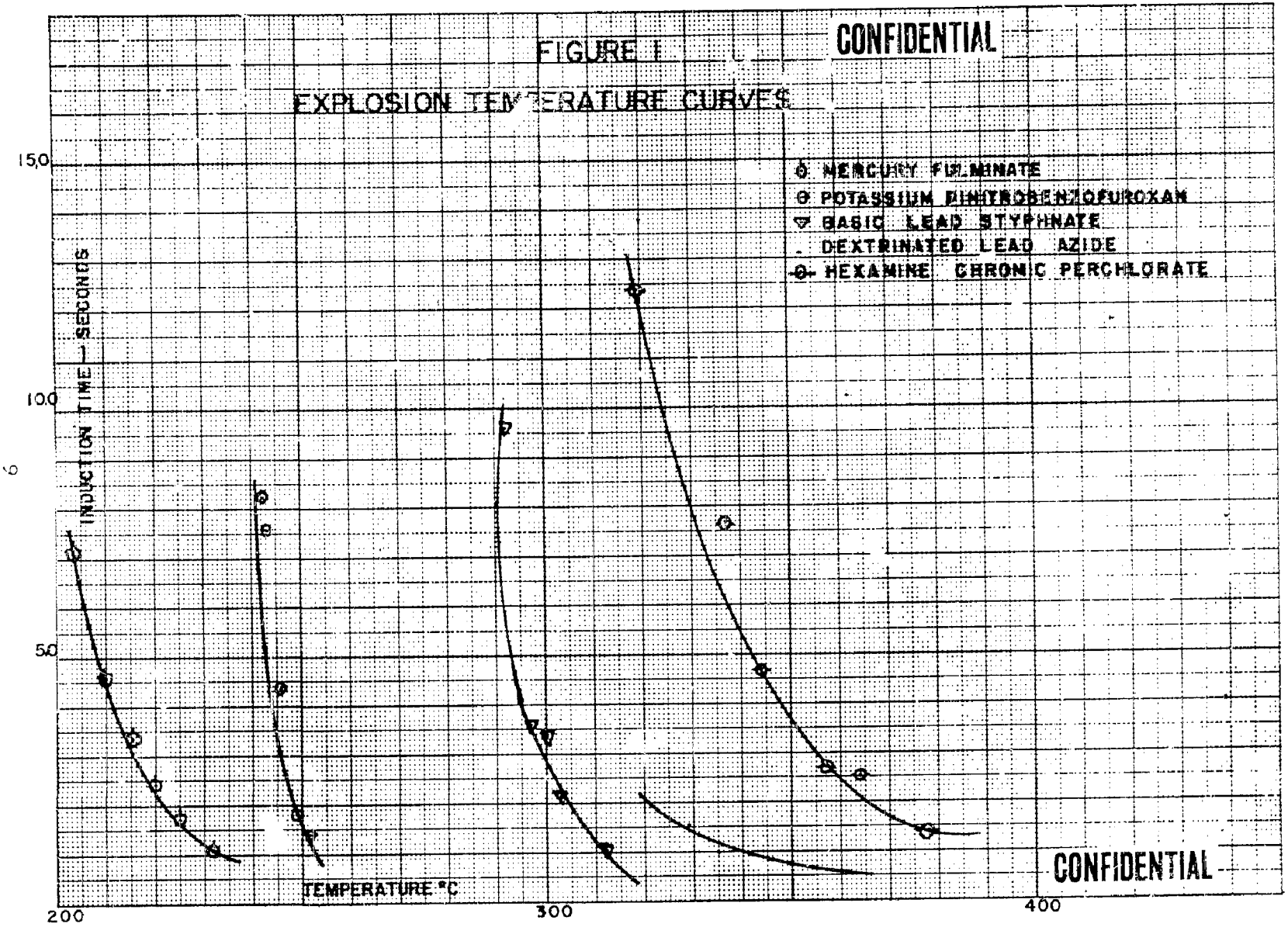

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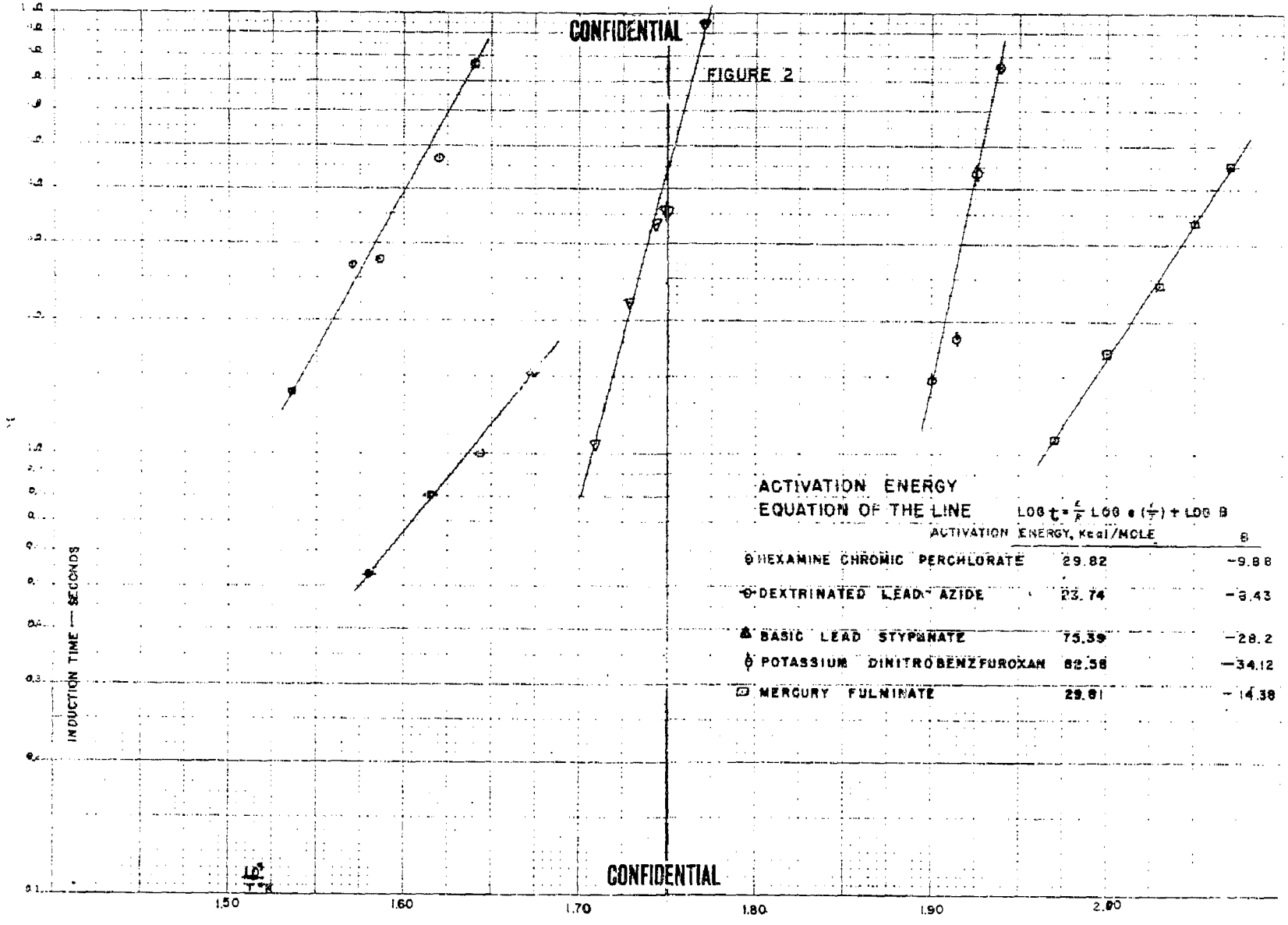
FIGURE 2



ACTIVATION ENERGY

EQUATION OF THE LINE $\log t = \frac{E}{R} \log e \left(\frac{1}{T}\right) + \log B$

	ACTIVATION ENERGY, Kcal/MOLE	B
⊖ HEXAMINE CHROMIC PERCHLORATE	29.82	−9.88
⊖ DEXTRINATED LEAD AZIDE	23.74	−8.43
⊿ BASIC LEAD STYPHNATE	75.39	−28.2
ϕ POTASSIUM DINITROBENZFUROXAN	82.56	−34.12
⊡ MERCURY FULMINATE	29.61	−14.38

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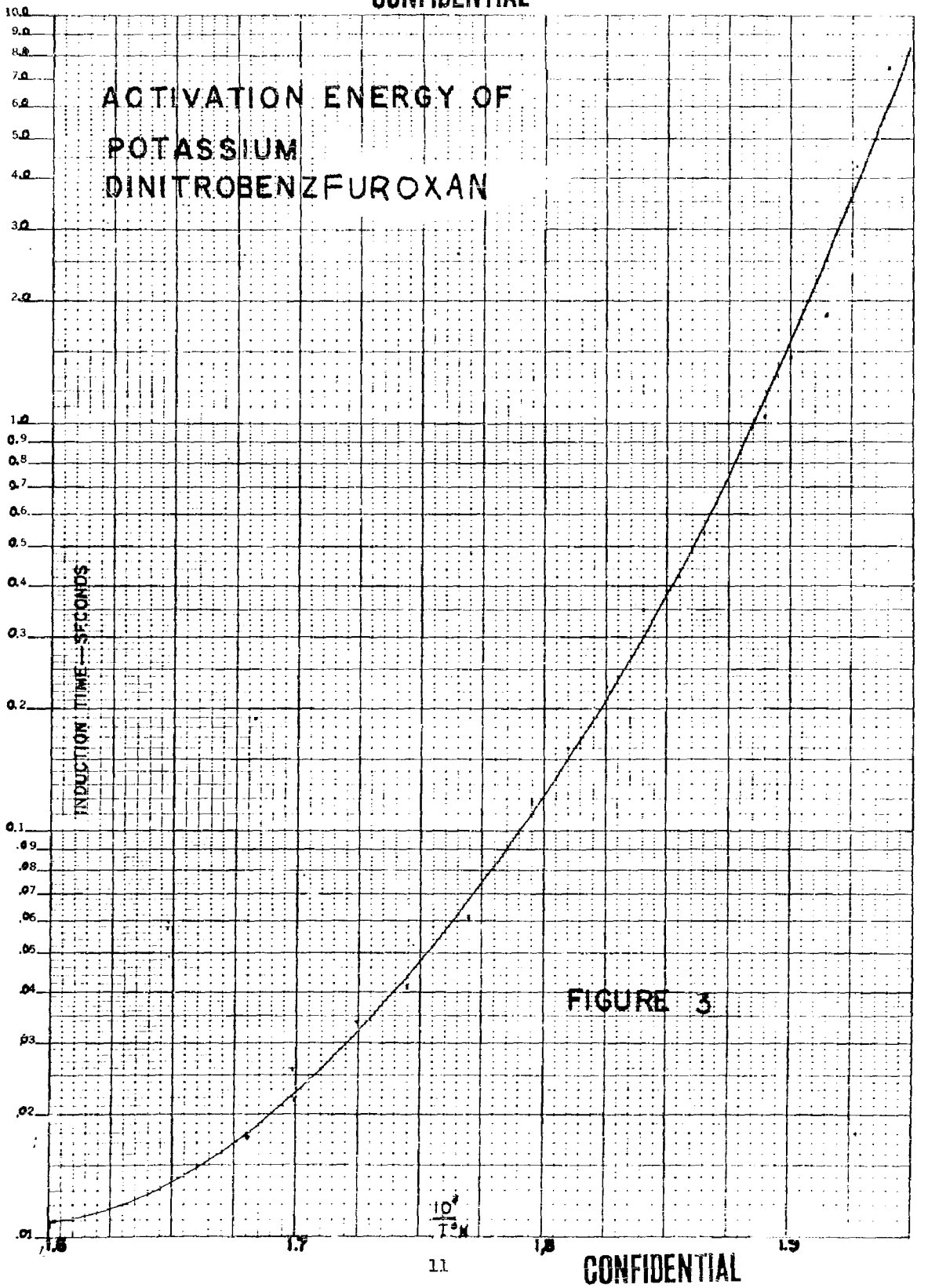
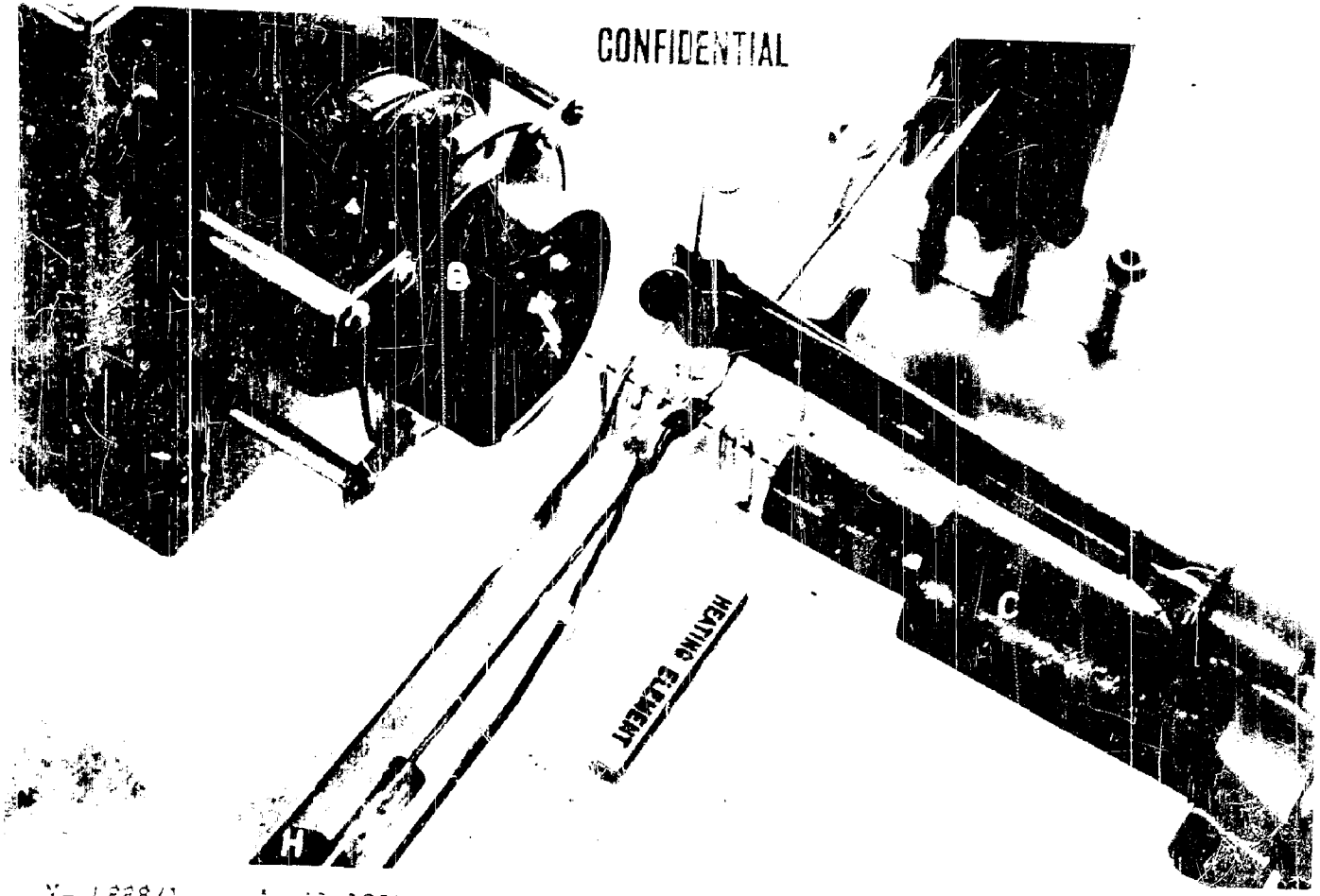

ACTIVATION ENERGY OF
POTASSIUM
DINITROBENZFUROXAN
INDUCTION TIME — SECONDS
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
.09
.08
.07
.06
.05
.04
.03
.02
.01
1.6
1.7
1.8
1.9
$\frac{10^4}{T°K}$

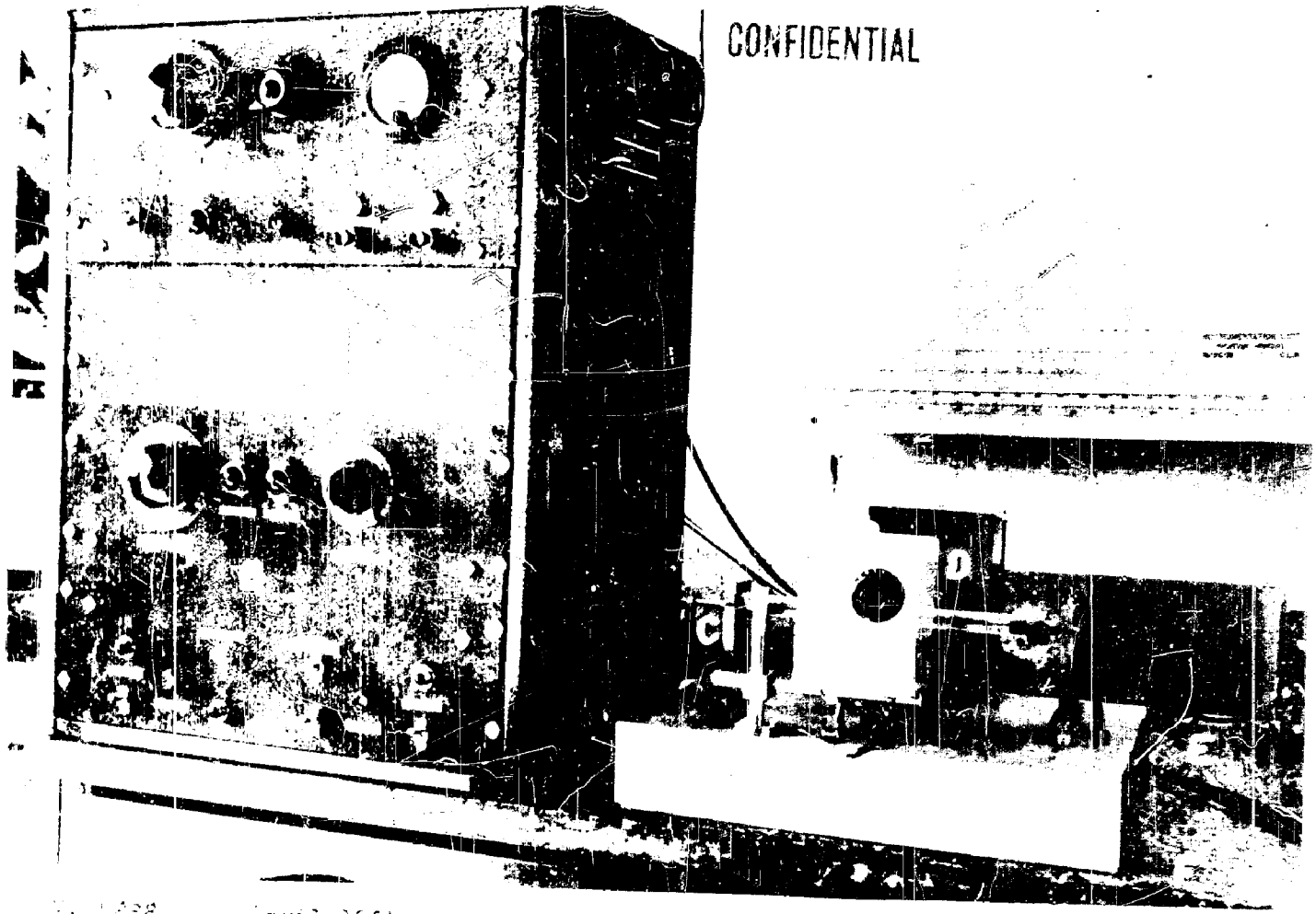
FIGURE 3



X-1228/1 April 1954 Picatinny Arsenal Plate I Ordnance Corps

Ignition Plate and Optical Photomultiplier Timing System

A - Path of light.
B - Light source through lens system.
C - Tube to Photomultiplier.
E - Electrically heated Ignition Plate
F - Wire carrying current to Ignition Plate.
F - Iron - Constant Thermocouple from center of Ignition Plate to Potentiometer.
G - Funnel to drop compound on plate.
H - Glass tube.



April 1954 Picatinny Arsenal Plate II Ordnance Corps

Ignition Temperature Apparatus

- Current Control Unit
- Control for Sensitivity Response of Photomultiplier
- Current Lead to Ignition Plate
- D - Photomultiplier.
- E - Thermocouple to Potentiometer.
- F - Lead to Timing Unit(Counter Chronograph).

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